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# Determination of Density Change of Glass by the Sink-Float Method. (VII) : Precision of Density Measurements

AUTHOR(S):

Mine, Masao; Nobusawa, Torao; Kawabata, Ayako

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obtained show that Traube's rule holds for the non-aqueous systems and the correlation between the surface tension depression of the solution ( $F$ ) and the mole fraction of the solute in the bulk phase ( $C$ ) can be expressed accurately by the equation

$$F = nkT \log \left( \frac{C}{\alpha} + 1 \right)$$

where  $k$  is Boltzmann's constant,  $T$  is the absolute temperature,  $n$  is a surface chemical constant giving the total number of sites of adsorbed molecules in a monolayer per unit area of the surface when no interaction between the adsorbed molecules exists, and  $\alpha$  is another surface chemical constant relating to the adsorption energy of the solute molecule on the solution surface. The values of  $n$  and  $\alpha$  determined from the experimental data are indicated in Table 1.

Table 1. Values of  $n$  and  $\alpha$ .

	$n$		$\alpha$	
	$E$	$N$	$N$	$E$
Methyl alcohol	$4.02 \cdot 10^{14}$	$5.32 \cdot 10^{14}$	$365.0 \cdot 10^{-3}$	$351.0 \cdot 10^{-3}$
Ethyl "	3.38 "	4.08 "	114.4 "	224.0 "
<i>n</i> -Propyl	2.42 "	3.21 "	50.0 "	153.0 "
<i>n</i> -Butyl	2.01 "	2.70 "	16.6 "	111.7 "
<i>n</i> -Amyl "	1.73 "	2.06 "	7.7 "	41.3 "

$E$  and  $N$  denote the cases where the solvents are ethylene glycol and nitrobenzene, respectively.

## 12. Determination of Density Change of Glass by the Sink-Float Method. (VII)

### Precision of Density Measurements

Masao MINE, Torao NOBUSAWA and Ayako KAWABATA

(Sawai Laboratory)

For some years, determination of glass densities with the sinkfloat method for the purpose of the composition control have been utilized successfully by many glass manufacturing factories in Japan. The authors applied the method to measure small variations in glass density for studying the configurational changes of glass during heat treatments in the annealing range (*This Bulletin*, 20, 54 (1950); 24, 71; 25, 70 (1951); 28, 60 (1952)). The water solution of potassium mercuric iodide was found sufficiently available as the buoyant liquid (*ibid.*, 19, 52, (1949)). In this country, thereafter, mainly the water solution of the complex salt have been used for the composition control of glass in factory practice. To investigate the precision of the method,

the relation between the reproducibility of the measurements and the heating rate of the water bath in the settling method was studied. Results obtained with the bromoform-mixture (density: ca. 2.5 g./cm.<sup>3</sup>) are summarized in Table 1, and compared with data by R. D. Duff (*J. Amer. Ceram. Soc.*, **30** (1) 12, (1947)).

Table 1. Comparison of Precision of Measurements.

Solution	s-tetrabromoethene <sup>1)</sup> -isopropyl salicylate		bromofordibutyl phthalate		
Temp. Coeff. of Density	-0.0019 g./cm. <sup>3</sup> /°C		-0.00212 g./cm. <sup>3</sup> /°C		
Heating Rate (°C/min)	0.1	-0.1 <sup>2)</sup>	0.05	0.1	0.2
No. of Samples	10	10	9	8	10
No. of Observations	10	20	10	10	10
$\sigma$ mean (°C)	0.090	0.074	0.013	0.047	0.104
$\sigma$ max. (")	0.138	0.094	0.027	0.067	0.152
$\sigma$ min. (")	0.057	0.050	0.001	0.025	0.042

1) Duff (l.c.).      2) Cooling.

The standard deviations ( $\sigma$ ) of the measurements of the settling-temperatures of glass samples increase rapidly with the heating rate of the bath. M. A. Knight (*ibid.*, 28 (11) 297 (1845)), using s-tetrabromoethene- $\alpha$ -monobromonaphthalene as the buoyant liquid and heating with the constant rate of 0.1°C/min, reported that, the standard deviation of the settling method is ca. 0.0001 g./cm.<sup>3</sup>. Though we could not utilize the automatic regulator to secure the constant rate of heating as he used, our result with the same heating rate agreed well with his. The precision of our measurements also, was not worse than that of Duff's (*l. c.*).

The relation between the heating rate of the bath and the reproducibility of measurements with the water solution of the complex salt (density: ca. 2.5g./cm.<sup>3</sup>) is summarized in Table 2-a).

Table 2. Heating Rate of Bath and Standard Deviations of Measurements of Settling Temperatures with K<sub>2</sub>HgI<sub>4</sub>-Water Solution.

a) Density of Solution: 2.4-2.6 g./cm.<sup>3</sup>.

Temp. Coeff. of Density: -0.00121 g./cm.<sup>3</sup>/°C.

Samples: 10,      Observations: 10.

Heating Rate (°C/min.)	0.05	0.1	0.2	0.4	1.0
$\sigma$ mean (°C)	0.064	0.036	0.059	0.093	0.160
$\sigma$ max. (")	0.069	0.097	0.114	0.147	0.246
$\sigma$ min. (")	0.027	0.001	0.011	0.023	0.071

b) Density of Solution: 3.0-3.1 g./cm<sup>3</sup>.  
 Temp. Coeff. of Density: -0.00147 g./cm.<sup>3</sup>/°C.  
 Samples: 11, Observations: 10.

Heating Rate (°C/min.)	0.1	1.0
$\sigma$ mean (°C)	0.102	0.281
$\sigma$ max. (")	0.155	0.488
$\sigma$ min. (")	0.062	0.151

The standard deviations of measurements with the water solution also increase approximately linearly with the heating rate, but its increase is far less than that of the bromoform-mixture. Then it can be said that, the water solution is more convenient than organic heavy liquids for rapid determinations at some expense of precision in density measurements.

As the densities of pure *s*-tetrabromoethane and bromoform are less than 2.9 g./cm<sup>3</sup>, these organic bromo-compounds are not applicable to density determinations of most lead-containing glasses of industrial uses (density: 3.0-3.1 g./cm.<sup>3</sup>). The water solution of the complex salt, on the other hand, having the maximum density of ca. 3.2 g./cm.<sup>3</sup>, is available for measurements almost all glasses for industrial purpose, by adjusting the density of the solution simply by evaporation or dilution. However, the solution of high concentration, having high viscosity, makes the measurements appreciably, inaccurate even if the same rate of heating of the bath is secured. The precision of measurements with a concentrated solution (density: 3.0-3.1 g./cm.<sup>3</sup>) is indicated in Table 2-b). Moreover, as the water solution is more stable to light, moisture and impurities than the organic bromo-compounds, it can be concluded that, the former is more favourable for factory practice of glass industry in this country.

### 13. Heat Resisting Enamel Coatings to be Applied on Nickel-Chrome Stainless Steel

Megumi TASHIRO and Hironori TERANISHI

(Sawai Laboratory)

As stated before (*This Bulletin*, 23, 6 (1952)) the heat resisting enamel to be applied on nickel-chrome stainless steel should give the coating of high expansion, while, at the same time, it should be difficult fusible.

Obviously, however, it would be difficult to find out the composition satisfying simultaneously the both seemingly contradicting characteristics. Although, for example, it is easy to get a large size in expansion by substituting silica with alkali etc., the fusibility of frit decreases as the necessary result.